## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF: George

Serial No.:

10/011,659

Filed:

11/29/01

**Group Art Unit:** 

1772

Title:

Nanocomposite reinforced polymer blend and method for

blending thereof

**Examiner:** 

Walter B. Aughenbaugh

## **DECLARATION UNDER RULE 132**

## I, Daniel Roberts, do hereby declare and say:

- 1. My home address is 2952 Bay Village Circle, Apt. 2064, Santa Rosa, CA 95403.
- 2. My current occupation is as an Engineering Consultant.
- 3. I am not employed by the applicant or their legal representative, other than in a consulting capacity to render technical advice for the purpose of this affidavit.
- 4. I received a B.S. in Chemistry from the University of Wisconsin in 1974.
- I received a PhD. in Polymer Science from the Massachusetts Institute of Technology in 1981, having written my Ph.D. thesis on "Fracture mechanics of polyethylene pipe materials".
- 6. I have more than 23-year's industrial experience as Senior Reliability Engineer (JDS Uniphase Corporation), Managing Engineer (Failure Analysis Associates, Inc.) Materials Development Manager/Principal Scientist (Electrical Products Division) Raychem Corporation, Senior Materials Engineer (Raynet Corporation, a Raychem Subsidiary), Project Manager, Senior Materials Scientist (Aerospace Division/Telecommunications Division, Raychem Corporation), and Research

- Chemist (Dow Chemical Co.), including two years as a consultant and expert witness.
- 7. During my industrial and academic career, I have developed specific expertise in the fields of the chemical, physical and electrical properties of polymers, and in particular, the properties of polymer blends and filler reinforced polymers, as well as the fracture mechanics of materials.
- 8. I have authored more than 26 papers in the above fields.
- 9. I have been a member of the following professional societies: American Chemical Society; American Ceramic Society; Society of Plastics Engineers; Plastics and Rubber Institute; Telecommunications Industry Association (TIA); Institute for Electrical and Electronics Engineers; The Society of Photo-Optical Instrumentation Engineers.
- 10. I have personal knowledge of what has been generally known to those of ordinary skill in the art of developing and compounding combinations of polymers and fillers from about 1974 to present.
- I have read U.S. patent application No. 2003/0099798 A1, U.S. Patent No. 6,270,477 B1 and US Patent No. 5,578,672.
- 12. I conclude that US Patent 6,270,477 B1 taken as a whole teaches that it is desirable to deploy a harder polymer in the body of the catheter tubing, using a softer polymer, copolymer or blend of polymers to lower the elastic modulus of the tip, hence making it more flexible.
- 13. I conclude that statement in US Patent 6,270,477 B1, at column 9, line 18-19 that "Different materials might also be combined or blended to select for desirable flexibility properties." would not, when considering the teachings of this application as a whole, suggest or provide a motivation that one can or should blend a reinforcing filler with one polymer to increase the modulus thereof if the intended use is for catheter tubing. On the contrary, in light of the teaching that one should use a polymer of lower elastic modulus at the tip, the motivation provided to one of ordinary skill in the art would be to combine or blend a lower

- modulus polymer with a higher modulus polymer for the portion of the tubing that is to become the tip of the catheter.
- 14. I conclude that US patent 5,578,672 does not teach or suggest that intercalated fillers, also described as nanocomposites by some, will improve the dimensional stability during the extrusion of type of thin wall tubing described in US patent application No. 2003/0099798 A1, but rather the reference to improvement in the '672 patent refers to the dimensional stability of a finish part or product at a higher temperature, as is generally measured by the Heat Deflection Temperature. Although the term dimensional stability can be a vague term, in the context of this patent it refers to that of the finished product, because of its application with respect to properties of the fabricated film product.
- 15. The statement found in US patent 5,578,672 at column 21, line 24 to line 26 would be interpreted by one of ordinary skill in the art as a general expression of a utility for intercalated fillers, and not specifically create the expectation that a particular combination of two or more mechanical properties can be expected to simultaneously improve, or that one property can be improved without a trade-off in another property.
- 16. I find that the property of an intermediate elastic modulus, between that of the components of the polymer blend, yet without a loss of tensile strength and toughness, and in some instances increased toughness, as obtained by the inventors as described in US patent application 2003/0099798 A1, would be unpredictable to one of ordinary skill in the art of polymeric materials development at the time the application was filed, that is as of November 29, 2001.
- 17. More specifically, in support of my opinion with respect to the ability to achieve an intermediate elastic modulus (without a loss of tensile strength and toughness), EXHIBIT 1, attached hereto, is a figure to illustrate the general range of experience when two polymers, or a polymer and an inorganic filler are blended together, wherein the compositional ratio is indicated on the X-axis, ranging from 100% component X on the left and 100% component Y on the right, with the

modulus or toughness as represented on the Y-axis. Curve A represents expected properties under the generic rule of mixing, in that over the composition range the resultant property is intermediate between that of the pure components, in linear proportion thereto. Real materials invariably produce either a positive or negative deviation from Curve A, being either a negative deviation, the "-" curve, wherein the resultant properties are somewhat less than the average, or a positive deviation, "+", wherein the property, the toughness or the modulus are greater than the average of the two components. It is very difficult to predict the nature of the deviation, as it can frequently depend on the quality of the dispersion of the components, during compounding, as well as the conditions during processing, such as extrusion. EXHIBIT 2 is a figure that shows the general behavior disclosed in the patent application when one blends 5% nanoparticles with either Nylon or pure PEBAX, which is the modulus, E, increases, whereas the tensile strength (TS) goes down. This is contrary to the rather generalized and nonspecific teaching of Beall, which suggests that the both the modulus and ultimate tensile strength can both be increased, or the impact strength can be increased. Consequently, given the consideration of EXHIBIT 1 in light of EXHIBIT 2, the expected result of blending Nylon and PEBAX that each contain nanocomposite filler has been illustrated by the figure of EXHIBIT 3. The bold dashed lines labeled E, represents the elastic modulus, and the bold dot-dashed line labeled TS. represents the tensile strength. In this hypothetical graph, the percentage nanocomposite is a constant 5%, the percentage PEBAX varying from 0 to 100% from the left side to the right side of the x-axis. Accordingly, one of ordinary skill in the art of developing polymer formulations would expect that the modulus and toughness would be an intermediate value across the composition range, as shown by the bold lines. Further the expected modulus of blending just Nylon and PEBAX is represented by the lighter weight dashed line in this figure. As the actual experimental data in the patent application at issue is largely at 2.5% nanocomposite loading in a 1:1 blend of PEBAX and Nylon, I have marked the expected properties of these compositions by a circle, for modulus, and a square. for tensile strength. One of ordinarily skill in the art would expect the 2.5%

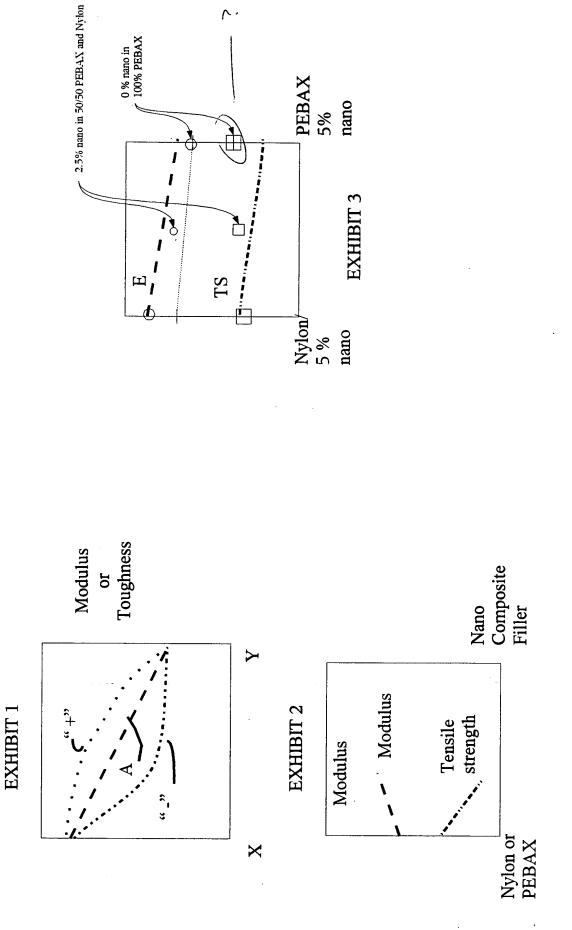
composition to follow the trend illustrated in EXHIBIT 2, wherein the modulus increases with an increase in percentage nanocomposite filler, and the tensile strength decreases. Thus, at the 50/50 composition, the circle is placed slightly above the lighter dotted line, representing a mid-point between 0 and 5% nanocomposite filler. In contrast, the square is placed slightly above the bold dotdashed line, which represents the expected tensile strength of the blends with 2.5% nano-composite, as a slightly higher tensile strength is expected when the 50/50 blends of just Nylon and PEBAX has only 2.5% nanocomposite filler. The figures in EXHIBIT 4, 5 and 6 combine selected data from FIG. 7 and FIG. 3 of the patent application to plot the actual mechanical properties for the three compositions indicated by the circles and squares in EXHIBIT 3, comparing the difference between blending of Nylon 11 and Nylon 12 with PEBAX. Specifically, referring first to the figure in EXHIBIT 4, it can be seen the modulus remains within the predicated range of EXHIBIT 3, for the intermediate composition (2.5 % Nanocomposite particles in both Nylon 11 and Nylon 12). However, with reference to EXHIBIT 5 the tensile strength behaves in a manner not expected, that is for Nylon 12, the tensile strength, at 12kpsi, is greater than would be predicted for the intermediate composition, greater than that of pure PEBAX as well as that of the blend of Nylon 12 and 5% nanocomposites. Whereas, for the Nylon 11 based blend the intermediated blend of 2.5 % nanocomposite is far inferior than the prediction of EXHIBIT 3, being less than expected (at 5.1 kpsi) than both the Nylon 11 5% nanocomposite mixture and the pure PEBAX. Further, the toughness, plotted in figure of EXHIBIT 6, as represented by the product of the tensile strength (TS) and strain to break (E) follows the same trend as the tensile strength. The results described in EXHIBIT's 4, 5 and 6 for Nylon 12 are similarly found for the Nylon 6 based compositions in FIG.8 of the patent application. Accordingly, I conclude that the beneficial properties of an intermediate elastic modulus, between that of the components of the polymer blend, yet without a loss of toughness from the pure unfilled polymer with the lower modulus (and in some instances increased toughness for selected compositions) described in US patent application 2003/0099798 A1, are not

predicable or expected despite the generalizations made in the Beall patent. Neither is such a result predicable or expected from what was commonly known to one of ordinary skill in the art of polymeric materials at the time the application was filed, November 29, 2001.

- 18. I find that the property improvements of 1) a lower propensity to retain dirt, and 2) an improvement in the dimensional stability in the process of extruding narrow diameter, thin wall tubing, as obtained by the inventors as described in US patent application 2003/0099798 A1, would not be expected from the references cited by the examiner to one of ordinary skill in the art of polymeric materials development at the time the application, November 29, 2001.
- 19. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001, and that such willful false statements may jeopardize the validity of the application or any patent issued there from.

Daniel Roberts, Ph.D.

Signature: Danie PR. Rolling Date: 9/24/04



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